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COMPLETE SPECIFICATION

Blend of Microcrystalline Wax and Olefin Polymer and Method for Production Thereof.

We, PHILLIPS PETROLEUM COMPANY, a corporation organised under the laws of the State of Delaware, United States of America, of Bartlesville, Oklahoma, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to blends of microcrystalline wax with high density, highly crystalline olefin polymers such as polyethylene. This invention also relates to a method for improving processability of polyethylene and copolymers of polyethylene prepared in the presence of a catalyst at comparatively low pressures.

Polyolefins, especially polyethylene, have long been used in casting and moulding operations. However, the polyolefins available until recently have been somewhat limited in applications where high temperatures were involved due to their relatively low softening points. It has recently been found that high softening point, high density, highly crystalline olefin polymers can be prepared at comparatively low pressure in the presence of certain catalysts. These polyolefins, especially polyethylene, show many superior physical properties over the older polyolefins, such as high softening point and high tensile strength and can be subjected to sterilizing heat without deformation. Although these polymers have had limited use in respect of certain applications for which they are otherwise ideally suited, the limited use being principally due to their very high melt viscosity. The very high melt viscosity of these highly crystalline olefin polymers present difficulties when the polymers are employed in extrusion or injection moulding processes. The high melt viscosity causes a reduction in flow rate to

such moulding machines, and frequently contributes to the production of mouldings having unsatisfactory gloss and smoothness.

According to the present invention there is provided a mouldable wax-olefin polymer or co-polymer blend which comprises 1 to 50 per cent. by weight, of microcrystalline wax and 50 to 99 per cent. by weight of a polymer or copolymer of a 1-olefin, said polymer having a density of at least 0.94 and a crystallinity of at least 75 per cent., and said copolymer having a density of at least 0.92, and a crystallinity of at least 75 per cent.

The present invention further provides a method for preparing a mouldable wax-olefin polymer or copolymer blend which comprises blending 1 to 50 per cent. by weight of microcrystalline wax with 50 to 99 per cent. by weight of a polymer or copolymer of a 1-olefin, said polymer having a density of at least 0.94 and a crystallinity of at least 75 per cent. and said copolymer having a density of at least 0.92, and a crystallinity of at least 75 per cent., and heating the blend to above the softening temperature of the polyolefin or olefin copolymer.

A procedure for determining crystallinity by nuclear magnetic resonance may be found in the Journal of Polymer Science, Volume 10, page 503 (1953) by Wilson and Pake.

While it is known to extend polyethylene with various waxes and other extenders and while it is known that certain waxes impart improved processability to various polymers, such blends of waxes with polyethylene in the past have been accompanied by degradation of the physical properties of the polymer. It is therefore surprising to find that when the microcrystalline wax is blended with these highly crystalline, high density polyethylenes, that the effect on the tensile strength of said polymer as well as other physical properties is only slight. That

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is, these polymers can be blended with the microcrystalline wax with only slight effect on the physical properties of the polymer. The blends of the present invention can be
5 extruded or injection moulded to form moulded articles having excellent gloss and smoothness.

The microcrystalline wax can be blended with the polymer in a variety of ways. The
10 two materials can be milled together on a roll mill or a Banbury mixer, for example, or they can be blended together in an extruder. Other methods of blending include dry blending and solvent blending. Numer-
15 ous variations of these methods can also be employed. For example, a solution of waxes can be admixed with a solid polymer, or a solution of wax can be mixed with a solution of polymer, followed by copre-
20 cipitation. The preferred method for blending the two materials together is to mill the two materials together at a temperature above the softening point of the polymer.

The main advantage of microcrystalline
25 wax over other materials which have been included in olefin polymers, is that the microcrystalline waxes have a limited compatibility with the polymer. By this, it is meant that the microcrystalline wax is not of
30 sufficiently high compatibility so as to provide no lubricating effect, yet they have a sufficient compatibility not to "bleed out" of the polymer.

The amount of microcrystalline wax
35 which is blended with the ethylene homopolymers and copolymers is in the range of from 1 to 50 weight per cent. based on the resulting blend. The amount of wax employed is dependent upon the molecular
40 weight of the polymer with which the wax is to be blended, and also, the amount of wax is dependent upon the desired ultimate use of the blend. For example, if
45 wax is to be blended with an olefin polymer of 50,000 molecular weight, the preferred amount of the microcrystalline wax in the blend is between 5 and 15 weight per cent. If the polymers are from 100,000 to 125,000
50 molecular weight, for example, the preferred amount of microcrystalline wax in the blend is between 10 and 25 weight per cent. The melting point of the wax which is selected will be dependent upon the ultimate
55 use of the blend, but for most uses, it is desirable to select the highest melting point microcrystalline wax, so as to maintain the softening point of the resulting blend as high as possible.

The wax-polymer blend of this invention
60 has several advantages; the wax-polymer blend has a considerably higher flow rate in injection and extrusion equipment than does the polymeric material alone and mouldings which are produced from the
65 blends of this invention have much smoother

surfaces and much higher gloss than do mouldings made from high molecular weight, high density, highly crystalline olefin polymers alone. It is believed that
70 the wax functions as a lubricant to improve the flow characteristics, but the process of blending a microcrystalline wax with an olefin polymer is inherently advantageous in that it provides a method for extending
75 a relatively expensive polymeric material with a relatively inexpensive microcrystalline wax.

The high density, highly crystalline olefin polymers which can be blended with the microcrystalline wax according to the
80 method of this invention, are ethylene homopolymers having a density of at least 0.94 and higher and a crystallinity of 90 per cent. and higher, and the copolymers of at least 50 weight per cent. ethylene with
85 other 1-olefins such as propylene, 1-butene and 1-hexene, which have a density of 0.92 and higher, and a crystallinity of 75 per cent. and higher.

A preferred and known method for pre-
90 paring such highly crystalline olefin polymers involves polymerizing 1-olefins having a maximum of 8 carbon atoms per molecule and no branching nearer the double bond than the 4-position either alone or
95 with other olefins by contacting same with a solid catalyst containing as an essential catalytic ingredient, chromium oxide, associated with at least one porous oxide selected from silica, alumina, zirconia and
100 thoria. Suitable olefins are ethylene, propylene, 1-butene, 1-pentene, 1-hexene, 1-octene, 4-methyl-1-pentene, 4-methyl-1-hexene, 4-ethyl-1-hexene, 6-methyl-1-heptene, 5-methyl-1-heptene and the like. These
105 materials can be polymerised alone or in admixture with each other to obtain solid or semi-solid polymers. Also, these olefins can be polymerized with other aliphatic olefins, such as butene-2 and butadiene. It is preferred that the chromium content of the catalyst be within the range of 0.01 to 10 weight per cent. and is highly preferable that an appreciable proportion of chromium
110 e.g., at least 0.1 weight per cent. of the catalyst be in the hexavalent state. The catalyst is finely divided and can be micro-spheroidal although the catalyst having particle sizes of up to 40 mesh can be employed satisfactorily. A highly desirable
115 catalyst is a chromium oxide catalyst associated with at least one additional oxide of the type already mentioned. A catalyst often preferred is one in which the oxide, or oxides, other than chromium oxide, have
125 been treated with fluoride, e.g., a volatile fluoride, such as hydrogen fluoride, followed by heating to remove residual volatile fluoride. A further improvement can be effected by incorporating a stabilizing
130

amount of strontium oxide in the catalyst.

The catalyst can be maintained in suspension in the reaction mixture by any suitable agitation means. The reaction temperature is preferably in the range 250 to 375°F. although temperatures outside this range can be used, for example at a temperature below the solution temperature of the polymer, e.g., as low as 150°F. or even lower so that the polymer is formed as discrete particles. In either case, solution or diluent process, the pressure will be sufficient to maintain the hydrocarbon diluent or solvent in liquid phase. For convenience, the solvent or diluent will hereinafter be referred to as diluent, since even when acting as a solvent, this hydrocarbon also serves as a diluent for the reaction. While vapour phase reaction can be employed, the present invention is described with a polymer prepared by solution polymerization. The reactor effluent is passed to a flash zone from which unreacted monomer is removed by flashing. In the solution process, the flashed material is frequently filtered to remove the catalyst. In the particle form process (polymer particles formed as discrete particles), the production per pound of catalyst is extremely high and generally no catalyst removal step is required. The polymer can suitably be recovered from solution or diluent by admixing the effluent with relatively cool water which results in polymer precipitation and subsequent separation of the precipitated polymer by steam stripping the polymer to remove hydrocarbon diluent after which the polymer is dried, remelted, extruded and cut into pellets. Other recovery methods can be employed, such as by solvent evaporation, cooling solvent to below the precipitation temperature of the polymer, etc. However, this is not part of the present invention and requires no discussion here.

Polyethylene produced by the process just described will ordinarily have a molecular weight in the range 35,000 to 100,000 or even higher, and in the case of particle form, for example, up to 200,000 or higher. Density will be in the range 0.95 to 0.97, e.g., approximately 0.96, and crystallinity will exceed 90 per cent. The tensile strength of the polymer as produced will ordinarily be of the order of 4,000 to 5,000 psi but can be higher or lower. The polymer ordinarily has a softening point of about 265°F. or higher. Polymers produced by this process have unsaturation which is predominantly of the terminal vinyl and/or trans-internal structure. So-called "branched-vinyl" unsaturation is substantially absent.

Another suitable, but less preferred and non-equivalent, method of preparing highly crystalline polymer is by polymerizing such olefins by contacting with a catalyst such

as a mixture of a compound represented by the formula AlR_3 , where R is a saturated aliphatic, cyclo-aliphatic or aromatic hydrocarbon radical or hydrogen; and a second component which is ordinarily a halogen compound of a metal such as titanium, zirconium, chromium or molybdenum. An example of such a catalyst is a mixture of triethylaluminum and titanium tetrachloride. Another suitable catalyst comprises a compound represented by the formula R_mAlX_n , where R is a hydrocarbon radical of the type previously described, X is a halogen and $m+n=3$, i.e. the valence of aluminium. Along with this latter type of catalyst, metal compounds, such as titanium dioxide and the tetraalkoxides of titanium, halides of titanium, as well as tetravalent titanium salts of organic carboxylic acids can be utilized. An example of such a catalyst consists of a mixture of diethylaluminum chloride, ethylaluminum dichloride, and titanium tetrachloride. A similar type of catalyst mixture comprises a halide of a group IVB metal, e.g., titanium tetrachloride and a free metal, such as metallic sodium or metallic magnesium. The polymerization reaction with these catalysts is ordinarily conducted at a temperature which can range from room temperature up to approximately 300°C. The reaction is preferably conducted with the olefin in admixture with a hydrocarbon such as isooctane, cyclohexane, or toluene, which is inert and non-deleterious to the catalyst under the reaction conditions. The pressures are ordinarily sufficient to maintain the inert hydrocarbon in substantially the liquid phase. The reactor effluent is ordinarily treated with a compound such as methanol, acetone, acetic acid, or water which decomposes the remaining catalyst and the polymer is recovered by vaporization of the hydrocarbon solvent or by precipitation of the polymer such as by cooling. Polymers produced by this general type of process will have molecular weight of the same order as those used in chromium oxide catalysts, a crystallinity of 80 to 85 per cent., and densities of 0.95.

While both of the foregoing types of polymerization can be carried out in batch processes, it is often preferred to carry out such processes continuously. In continuous processes, it is frequently preferred to carry out the reaction in a plurality of reactors in series. Continuous processes are within the skill of the art and need no further discussion here.

It will be noted that the foregoing specification as to density and crystallinity are not satisfied by most of the polyethylenes which have heretofore been available on the market. Most such polyethylenes have been produced by polymerizations at ex-

tremely high pressures, e.g., of the order of 10,000 psi or higher, usually in the presence of a peroxide type catalyst or without any catalyst. These materials ordinarily have densities of the order of 0.91 or 0.92 and crystallinities no higher than 60 per cent. in many cases. They ordinarily have a molecular weight within the general range 5,000 to 30,000 and tensile strengths of the order 1,500 to 2,000 psi. The unsaturation in such polymers is predominantly of the "branched vinyl" type.

As has been indicated, the polyolefin blended with the microcrystalline wax of this invention is an ethylene homopolymer or a copolymer of ethylene and one of the other 1-olefins indicated and wherein the resulting polymer has a crystallinity of at least 75 per cent. and a density of at least 0.94.

The superiority of moulded articles from these blends has been previously described. The wax-polymer blends of the present invention can be utilized in any injection moulding or extrusion moulding application. Some examples of such applications are fibres, films and moulded articles.

The microcrystalline waxes which can be employed in the blends of the present invention are those which melt between about 130 and 220°F. These waxes are referred to as microcrystalline waxes as opposed to paraffin waxes. Such microcrystalline waxes are well known in the art. Paraffin and microcrystalline waxes are both separated from crude petroleum. However, the process of manufacture and the resulting products are quite different.

As crude petroleum is subjected to distillation by heating in a still at atmospheric pressure, the following products are removed in the order of their increase in boiling point: light petroleum gases; gasoline; naphtha; kerosene; gas oil; paraffin wax distillate; light neutral lubricating oil fractions; and a residue in the still which will not distil overhead at atmospheric pressure without decomposition. Paraffin wax is separated from the paraffin wax distillate by a relatively simple process, while the microcrystalline wax which cannot be distilled without decomposition, is separated by a complex series of solvent separations from the residue remaining in the still. That is, the paraffin wax comes overhead during distillation whereas microcrystalline wax is to be recovered from the still bottoms.

Chemically, both paraffin and microcrystalline waxes consist of saturated hydrocarbons. Paraffin and microcrystalline waxes are both long chain compounds, but microcrystalline wax has much higher molecular weight. The common commercial paraffin waxes have molecular weights of 360 to

420, which means that the average molecule of these hydrocarbons contains 26 to 30 carbon atoms. The molecular weight of commercial microcrystalline waxes is 580 to 700. That is, the average molecule contains 41 to 50 carbon atoms. Extensive X-ray diffraction work indicates differences in the chemical structure of microcrystalline wax molecules and paraffin wax molecules in addition to a difference in their molecular weights. Paraffin waxes have been found to consist mainly of straight chain molecules whereas the microcrystalline wax has a high degree of branching. The branched chains in the microcrystalline wax are probably located at random along the carbon chain while any branching found in paraffin wax is near the end of the chain. Other differences between these two waxes are noted, for example, paraffin wax has very little affinity for oil whereas microcrystalline wax has an extremely high affinity. For example, the paraffin wax after recovery will have less than about 0.5 per cent. oil whereas the oil content of microcrystalline wax varies with the grade of wax but is usually within the range 2 to 12 per cent.

Those skilled in the art will know that antioxidants, dyes, fillers and the like can be present in the blend without departing from the scope of this invention.

Several examples are given in which a high density, highly crystalline polyethylene, prepared by the chromium oxide catalyzed polymerization, was blended with microcrystalline wax. These runs were made to illustrate the advantage of the wax-polymer blend of the present invention and they are in no way intended to limit the invention to the embodiment shown therein.

Example 1

Preparation of Ethylene Polymer

Ethylene was polymerized in a chromium oxide-catalysed solution polymerization at 291°F. and 420 psig. The ethylene was fed to the reactor continuously at the rate of 62 pounds per hour, while the solvent cyclohexane was fed to the reactor continuously at the rate of 337 pounds per hour. The catalyst, activated at 1,175°F. and containing 4.63 per cent chromium as CrO_3 and 0.05 per cent. Cr as Cr_2O_3 , was fed to the reactor so as to maintain a concentration of 0.24 per cent by weight catalyst in the reactor. The residence time in the reactor was 2 hours, and the productivity was 62 pounds of polymer per pound of catalyst. The polymer had a melt index of 0.90, which corresponds to a molecular weight of 40,800, a crystallinity as determined by nuclear magnetic resonance at room temperature of about 95 per cent. and a density of about 0.96 gms/cc.

Preparation of Microcrystalline

Wax-Polyethylene Blend

The desired amount of microcrystalline wax, a commercially available wax having a melting point in the range 180 to 185°F., and the desired amount of the above-prepared high molecular weight polyethylene were blended together in a Banbury mixer for 10 minutes at 310°F.

Evaluation of Wax-Polyethylene Blends

- 10 The above prepared blends were each charged to a 3 oz. Fellows injection moulding machine and injected into a spiral

mould, using mould conditions of 525°F. and 20,000 psig. The machine was set for the desired cycle time and allowed to "line out" by operating for approximately 20 minutes before samples were taken. By operating in this manner, the polymer in the machine can thus be of a uniform temperature. After the 20 minute "lining out" period, ten of the moulded spirals were collected, and the average weight of each spiral was determined. The results of these runs are expressed below as Table 1.

Table 1

Run No.	Wax/Poly-ethylene Ratio (wt.)	Weight of Spiral, Grams	% Increase over Poly-ethylene Alone	Tensile strength psi compression moulded	Elongation %	Environmental Stress Cracking (F50) Hours	Impact Strength ft. sq./inch per inch notch
1	0/100	1.48	—	4442	20	12-18	4.07
2	0.5/99.5	1.48	0	4489	19	Not mea-	4.11
3	1.0/99.0	1.48	0	4537	20	sured	5.80
4	3.0/97.0	1.60	8	4512	14	13	3.22
5	5.0/95.0	1.62	10	4562	13	14	2.54
6	10.0/90.0	1.72	17	4602	24	15	1.96
7	15.0/85.0	1.92	30	4325	17	10	0.86
8	20.0/80.0	2.00	35	4232	11		0.56

Example 2

A series of runs were made in which a very high molecular weight (128,000) ethylene polymer was blended with the microcrystalline wax of Example 1. These runs were carried out according to the following procedure.

Preparation of Ethylene Polymer

Two runs were made in which ethylene was polymerized in a pentane slurry at 210°F., after which the polymer was blended together. The polymerization conditions were the same for each of these runs, and are as follows:—

Conditions

- 40 Pressure 450 psig
 Temperature 210°F.
 Pentane Feed Rate 6167#/hr.
 Ethylene Feed Rate 85 ft³/hr.
 Catalyst Analysis 2.5% Cr as CrO₃
 45 Catalyst Activation Temperature 1175°F.
 Reactor Catalyst Concentration 0.169 wt.%
 Polymer Concentration in Reactor 20.0%

Polymer Properties

- | | Run I | Run II |
|--|---------|---------|
| 50 Density (gms/cc) | 0.954 | 0.958 |
| Softening Temp., °F. | 262 | 262 |
| Melt Point, °F. | 256 | 256 |
| Impact Strength ft./s/in. notch | 15.64 | 19.35 |
| 55 Environmental Stress Cracking (F50) Hours | 155 | 285 |
| Stiffness, psi | 170,000 | 139,000 |
| Crystallinity | 95% | 95% |

Blended Polymer Properties

- | | |
|--|---------|
| Molecular Weight (by inherent viscosity) | 128,000 |
| Ash | 0.84 |
| Tensile, comp. moulded, psi | 4,249 |
| Tensile, inject. moulded, psi | 7,376 |
| Elongation, comp. moulded, % | 82 |
| Elongation, inject. moulded, % | 7 |

The above-described blend of ethylene polymers was blended with the microcrystalline wax of Example 1 by the same procedure, and at the same conditions as described in Example 1. The wax-polymer blends were then injection moulded into spirals as described in Example 1. The results of these runs are expressed below as Table II. In this table, results are given both as actual and calculated. The calculated values were obtained by plotting the actual values and assuming that the change in properties with varying wax content was linear. A straight line was drawn through the largest number of points. The calculated values are the values on this line at a given wax-polymer ratio.

Table II

Table 11									
Run No.	Wax Polymer Ratio (wt.)	Spiral, Grams		Tensile Strength	Elongation %	Environmental Stress Cracking (F50), Hours	Impact Strength Ft. sq./inch notch		
		Actual	Calc.	psi Compression Moulded					
5	1	0/100	.65	.57	4249	82	155-285	15.6-19.4	70
	2	1/99	.60	.60	Not measured	Not measured	Not measured	Not measured	
	3	3/97	.61	.61	Not measured	Not measured	Not measured	Not measured	
	4	5/95	.90	.65	Not measured	Not measured	Not measured	Not measured	
	5	10/90	.74	.74	4062	42	17	3.78	
10	6	15/85	.80	.80	4078	27	17	3.79	75
	7	20/80	.92	.92	Not measured	Not measured	Not measured	Not measured	
	8	25/75	1.00	1.00	Not measured	Not measured	Not measured	Not measured	
	9	30/70	.96	1.08	Not measured	Not measured	Not measured	Not measured	

Example 3

15 A series of runs were made in which blends of the microcrystalline wax of the previous examples and the very high molecular weight ethylene polymer of Example 2 were extruded by means of a commercial screw-type extrusion machine. In these runs the desired blend was extruded through a 1 in. NRM bench extruder and the extrusion rate was determined. The extruder was maintained at 500°F. for each run, and the speed of the screw was constant for all runs. The results of these runs are expressed below as Table III.

Table III

Run Number	Wax/Polymer Ratio	Extrusion Rate Grams/min.	% Increase
30	1	0/100	18.5
	2	5/95	23.2
	3	10/90	27.6
	4	15/85	32.4
	5	20/80	31.9
35	6	25/75	23.8

Comparative Example

A series of runs were made wherein microcrystalline wax was blended with a commercially available high pressure polyethylene having a crystallinity of about 60 per cent. and a density of about 0.92. The data are shown below:—

Blend No.	Ratio	Components	Tensile Strength	Elongation	Spiral Flow, grams at 325°F. and 20,000 psi	
Control	—	DYNH*	2000	600	18.8	95
40	1	5/95 Wax-DYNH	1817	525	21.7	
	2	10/90	1363	412	23.3	
	3	15/85	1232	115	27.2	100

* A commercially prepared polyethylene having a crystallinity of about 65 per cent. and a density of about 0.92.

It will be noted that there is a marked decrease in tensile strength and also in elongation when increasing quantities of microcrystalline wax are added to the commercial polyethylene. Thus, the tensile strength drops from 2,000 psi for the pure commercial polyethylene to 1232 psi for a blend containing 15 per cent. wax. The elongation decreases from 600 per cent. to 115 per cent. at the same concentration. In Table I of Example I, it is shown that the tensile and elongation of a mixture of microcrystalline wax in the highly crystalline polyethylene shows only a very minor decrease with 15 per cent. wax, thus the tensile strength of the wax-free polyethylene is shown to be 4,442 psi and the blend containing 15 per cent. wax has a tensile of 4325. The elongations are 20 and 17 per

cent. respectively. The weight of material injected into a spiral mould increases for both blends when adding microcrystalline wax.

WHAT WE CLAIM IS:—

1. A mouldable wax-olefin polymer or copolymer blend which comprises 1 to 50 per cent. by weight of microcrystalline wax and 50 to 99 per cent by weight of a polymer or copolymer of a 1-olefin, said polymer having a density of at least 0.94 and a crystallinity of at least 75 per cent. and said copolymer having a density of at least 0.92 and a crystallinity of at least 75 per cent.

2. A blend according to Claim 1, wherein said 1-olefin polymer is a copolymer of at least 50 parts ethylene per 100 total parts and a second 1-olefin of 3 to 8 carbon atoms having no branching nearer the double bond than the 4-position.

3. A blend according to Claim 1 or 2,

wherein said 1-olefin polymer is polyethylene having a density in the range of 0.95-0.97 and a crystallinity of at least 90 per cent.

- 5 4. A method for preparing a mouldable wax-olefin polymer or copolymer blend which comprises blending 1 to 50 per cent. by weight of microcrystalline wax, with 50 to 99 per cent. by weight of a polymer or
10 copolymer of a 1-olefin, said polymer having a density of at least 0.94 and a crystallinity of at least 75 per cent., and said copolymer having a density of at least 0.92, and a crystallinity of at least 75 per cent.,
15 and heating the blend to above the softening temperature of the polyolefin or olefin copolymer.

- 20 5. A method according to Claim 4, wherein the polyolefin is a copolymer of a major proportion of ethylene and a minor proportion of a second 1-olefin of 3 to 8 carbon atoms having no branching nearer the double bond than the 4-position.

6. A method according to Claim 4, wherein the polyolefin is polyethylene having a density in the range 0.95-0.97 and a crystallinity of at least 90 per cent. 25

7. A method according to any one of Claims 4 to 6, wherein said blend is there- after moulded. 30

8. A method for preparing a mouldable wax-olefin polymer blend substantially as hereinbefore described with reference to the Examples.

9. A method for moulding a mouldable wax-olefin polymer blend substantially as hereinbefore described with reference to the Examples. 35

10. A mouldable wax-olefin polymer blend substantially as hereinbefore described with reference to the Examples. 40

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